PCT/GB2004/003529

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I also certify that the attached copy of the request for grant of a Patent (Form 1/77) bears an amendment, effected by this office, following a request by the applicant and agreed to by the Comptroller-General.

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Dated 20 August 2004

Patents Form 1/77

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18AUG03 E830857-1 B14991
P01/7700 0.00-0319269.7

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

The Patent Office

Cardiff Road
Newport
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1. Your reference

LCD1

2. Patent application number
(The Patent Office will fill this part in)

0319269.7

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

① ANTHONY BERNARD DAVEY, 30 ARGYLE STREET, CAMBRIDGE. CB1 3LR

② WILLIAM ALDEN CROSSLAND, 15 SCHOOL LANE, HARLOW, ESSEX. CM20 2QD

Patents ADP number *(if you know it)*

① 5522131002

② 2564159700(1)

If the applicant is a corporate body, give the country/state of its incorporation

4. Title of the invention

BISTABLE FERROELECTRIC LIQUID CRYSTAL DEVICES USING SILOXANE OLIGOMERS.

5. Name of your agent *(if you have one)*

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

~~DR. A. B. DAVEY.~~
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WC1R 4PJ

Patents ADP number *(if you know it)*

~~8679780400~~

6. Priority: Complete this section if you are declaring priority from one or more earlier patent applications, filed in the last 12 months.

Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

7. Divisionals, etc: Complete this section only if this application is a divisional application or resulted from an entitlement dispute (see note f)

Number of earlier UK application	Date of filing *(day / month / year)*

8. Is a Patents Form 7/77 (Statement of inventorship and of right to grant of a patent) required in support of this request?

Answer YES if:

a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.

Otherwise answer NO (See note d)

No

9. Accompanying documents: A patent application must include a description of the invention. Not counting duplicates, please enter the number of pages of each item accompanying this form:

Continuation sheets of this form	
Description	7
Claim(s)	
Abstract	
Drawing(s)	5 only

8

10. If you are also filing any of the following, state how many against each item.

Priority documents	
Translations of priority documents	
Statement of inventorship and right to grant of a patent (Patents Form 7/77)	
Request for a preliminary examination and search (Patents Form 9/77)	
Request for a substantive examination (Patents Form 10/77)	
Any other documents (please specify)	

11. I/We request the grant of a patent on the basis of this application.

Signature(s) A B Do [signature] Date 13.08.03

12. Name, daytime telephone number and e-mail address, if any, of person to contact in the United Kingdom

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Notes

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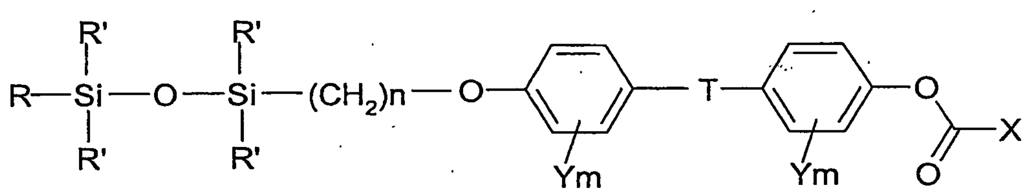
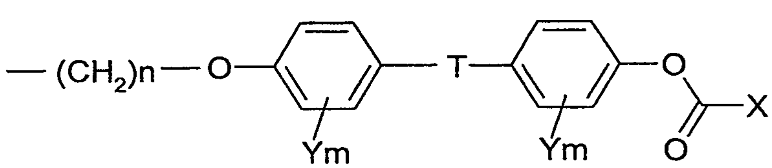
BISTABLE FERROELECTRIC LIQUID CRYSTAL DEVICES USING SILOXANE OLIGOMERS

[0001] Ferroelectric liquid crystal (FLC) devices are based on chiral smectic liquid crystals and are described by J. Dijon in Chapter 13 (pages 307-354) of "Liquid Crystals Applications and Uses, Vol. 1, edited by B. Bahadur, published by World Scientific Publishing co. Pte. Ltd (Singapore) in 1990. In particular cells arranged in bookshelf geometry are described on page 309. These are very thin cells, for example 1 to 10μm thick, arranged between alignment layers. The boundary conditions imposed by the alignment layers are strong enough to suppress the helix structure of the chiral smectic liquid crystal. The smectic layers are generally perpendicular to the cell plates but are capable of switching between two optical states which are at an angle (the tilt angle) to the perpendicular.

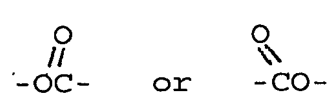
[0002] US-A-5498368 describes a siloxane- containing compound having the general formula



wherein R represents an alkyl group having from 1 to 10 carbon atoms or the group



each R' represents an alkyl group having from 1 to 4 carbon atoms, T is

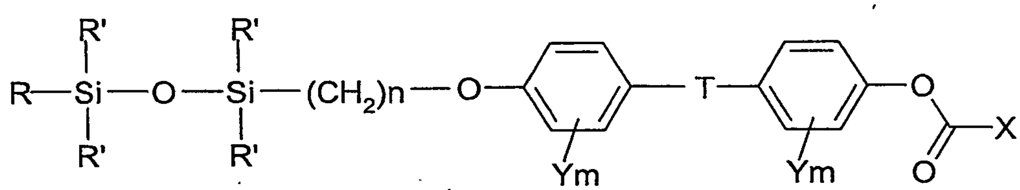


X represents an alkyl or halogen-substituted alkyl group having at least one chiral centre, Y represents a fluorine atom, m has a value of 0, 1 or 2 and n has a value of 10, 11 or 12.

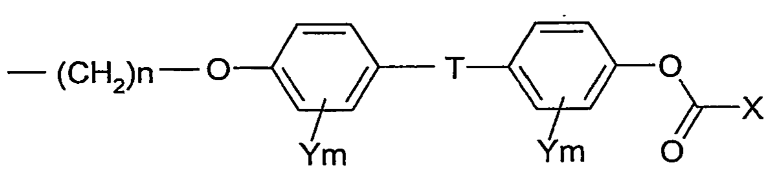
[0003] The ferroelectric liquid crystal siloxane oligomers of US-A-5498368 exist as neat compounds or in mixtures which exhibit relatively high tilt angles. Measurements of the tilt angle of other liquid crystal materials, as disclosed in JP-A-01-144491 have shown not only a variation from polymer to polymer but also a reduction of the tilt angle with increasing temperature. The tilt angles of the ferroelectric liquid crystal materials of US-A-5498368 are substantially independent of temperature over a broad temperature range including ambient.

[0004] We have found according to the invention that the ferroelectric siloxane oligomers of US-A-5498368 can be simply made to form a high quality bookshelf FLC structure that switches in a bistable fashion.

[0005] According to one aspect of the invention, a cell capable of switching in a bistable fashion comprises a siloxane-containing compound having the general formula



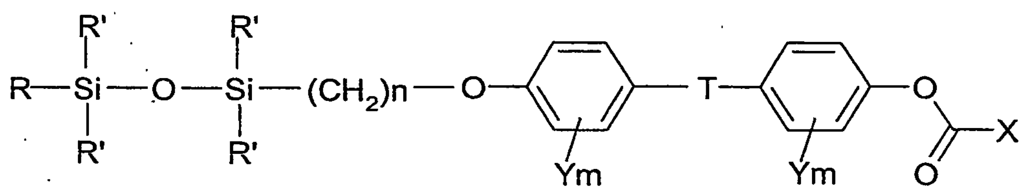
wherein R represents an alkyl group having from 1 to 10 carbon atoms or the group



each R' represents an alkyl group having from 1 to 4 carbon atoms, T is

X represents an alkyl or halogen-substituted alkyl group having at least one chiral centre, Y represents a fluorine atom, m has a value of 0, 1 or 2 and n has a value of 10, 11 or 12 arranged between alignment layers of polyamide or polyester.

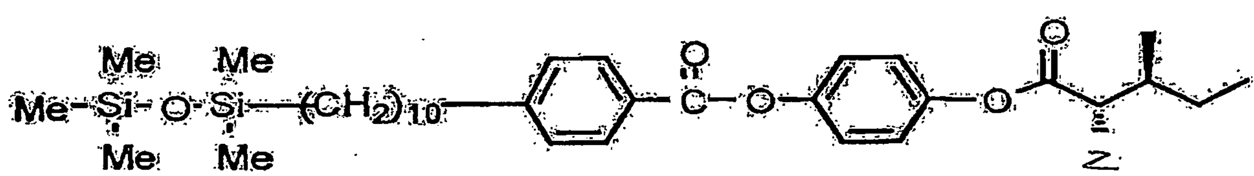
[0006] The invention also includes a process for bistable switching of a ferroelectric liquid crystal device, in which a cell comprising a siloxane- containing compound having the general formula



wherein R, R', T, X, Y, m and n are defined as above, arranged between alignment layers of polyamide or PET or PBT, is switched by applying an electric field of 2 to 50V per μm cell thickness at a frequency of 1 to 10KHz.

[0007] It is likely that such voltages could be made available in multiplexed FLC large panel displays or in liquid crystal on silicon (LCOS) devices so devices could be aligned, and if necessary restored in situ. This offers a new route to bistable switching, other than the conventional methods of either sheer alignment (which is not applicable to display manufacture) or cooling down to the smectic C phase via nematic and smectic A phases.

[0008] The ferroelectric liquid crystal siloxane oligomers of the invention do not have smectic A or nematic phases; the smectic C phase is formed directly on cooling from the isotropic state. The most preferred ferroelectric liquid crystal siloxane oligomers for use in the invention are those of the formula where Z is Cl or F.



[0009] The alignment layer is preferably of polyamide. The preferred polyamide is nylon-6,6, although nylon-6 is an alternative. Polyester alignment layers are also suitable, particularly an aliphatic/aromatic polyester such as a polyalkylene terephthalate, for example PET(polyethyleneterephthalate) or PBT (polybutyleneterephthalate). Polyimide alignment layers, which are the most commonly used alignment layers for liquid crystals, have not been found to allow bistable switching by application of an electric field alone without temperature change.

[0010] The structure of the cell can be as illustrated in schematic cross-sectional view in Figure 1 of US-A-5498368. The siloxane-containing crystal material (17) is interposed between a pair of substrates (10) which may be constructed of glass or a suitable polymer. The inner surfaces are successively coated with a transparent conducting film (12, 13) of indium tin-oxide and aligning agent (14, 15). The aligning agent (14,15) is preferably polyamide or alternatively PET or PBT in the cell of the present invention. Spacers (16) define the cell thickness. The conducting film may cover the whole inner surface of the substrate or may be etched into a suitable pattern.

[0011] Advantages of the invention include that no heating/cooling cycles are required to create the alignment required for bistable switching. Hence re-alignment can be carried out during the life of manufactured FLC displays if necessary. Display devices that have had their surface induced alignment damaged (e.g. by shock, deformation or temperature excursions) could be routinely repaired (e.g. during device start-up or shut-down) in service.

[0012] Since the materials do not have overlying smectic A or nematic phases, the tilt angle (and therefore the switching angle) is high and temperature independent. The formation of chevrons and related defects is not observed. Only such FLCs that exhibit the chiral smectic C through such a first order transition are capable of giving the 45 degree tilt angles that are necessary for efficient phase modulation. Previously there has been no practical method of making such materials truly bistable. The cells of this invention can be used for the display industry (with tilt angles near 22.5 degrees) as well as for real time holographic devices using phase modulation (with tilt angles approaching 45 degrees). The tilt angle

obtained can be varied by selection of the mesogen present in the ferroelectric liquid crystal siloxane oligomer of the invention

[0013] Electro-optic bistability is the basis of the remarkable ability of FLC displays to be passively multiplexed (i.e. to operate without positioning transistor circuitry at each pixel). Achieving robust bistable surface alignment has been a major uncertainty in developing large area FLC display panels. In LCOS displays which do have an active backplane which effectively speeds up the frame time we believe that only bistable FLC devices can have a continuously valid image. As far as we know all commercial FLC LCOS devices do not rely on bistability and hence have to "switch off" the illumination at intervals. As the cells of the invention are truly bistable, they can have a continuously valid image.

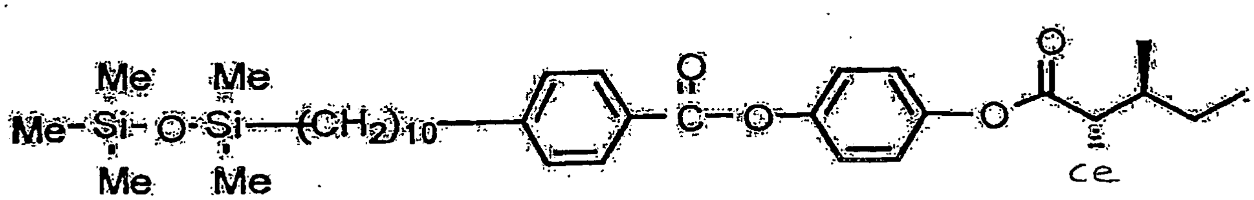
[0014] Bistability is also required for the use of FLCs in devices for storing or accumulating information, e.g. optically addressed spatial light modulators for ultra high resolution displays for holographic systems. Latching switches for telecommunications applications also need to be bistable.

[0015] Layer rotation of the bookshelf structure can be achieved by application of an electric field with an asymmetric waveform, as described by I.G.Magnolis et al in the paper “Control of the electro-optic bistability of some useful FLCs useful for binary phase optical modulators” in Mol. Cryst. Liq. Cryst., 351, (2000) at page 305. Layer rotation with retention of a truly bistable material has not previously been achieved.

[0016] The invention is illustrated by the following Example, which will be described with reference to the accompanying Figures.

Example

[0017] A ferroelectric liquid crystal siloxane oligomer of the formula



was assembled in a cell of the type shown in Figure 1 of US-A-5498368. The cell was 2μm thick. The alignment layers 14, 15 were rubbed polyamide layers 20nm thick. The cell was heated to above 53°C to convert the siloxane oligomer to the isotropic state and cooled to form the smectic C phase. An electric field of 20V at frequency 1KHz was applied.

[0018] Figure 1(a) shows the stability of the tilt angle at temperatures in the range 25 to 50°C.

[0019] Figure 1(b) shows the electrooptic response of the cell to an applied voltage varying between +7 and –7V.

[0020] Figure 2(a) is a photomicrograph of the siloxane oligomer in the cell before and after alignment by electric field, showing the bookshelf structure achieved after alignment. Figure 2(b) shows the structures before and after alignment of a commercial FLC aligned by prior art methods.

[0021] Figures 3(a) and (b) are photographs of light taken through the cell between crossed polarisers in the positively and negatively switched conditions. Figure 3(a) shows that light is transmitted in one state (the bright state) whereas Figure 3(b) shows that no light is transmitted in the other state (the dark state).

[0022] Figures 4(a) and (b) show asymmetric waveforms which can be used to induce layer rotation of the FLC in the cell, as described in the Mol. Cryst. Liq. Cryst. paper mentioned in Figure 4.

[0023] Figure 5(a) shows the voltage applied to the cell over time. Positive voltage is applied so that the FLC is aligned in its bright state followed by no voltage then negative voltage. Figure 5(b) shows the response of a photodiode to light from crossed polarisers passing through the cell. The response is positive (light is transmitted) after application of the

positive voltage and substantially stable while no voltage is applied and drops to zero when a negative voltage is applied.

[0024] Figure 5(c) also shows the voltage applied to the cell over time. In this case negative voltage is applied so that the FLC is aligned in its dark state followed by no voltage then positive voltage. Figure 5(d) shows the response of the photodiode. No response is recorded (no light is transmitted).

Further consequences of the SmC* to isotropic Transition.

CDRR8: Room Temperature $\longrightarrow$ SmC* $\xrightarrow{57\,°C}$ Iso

High tilt angle, independent of temperature.

CDDR8 –
Tilt angle Vs Temperature

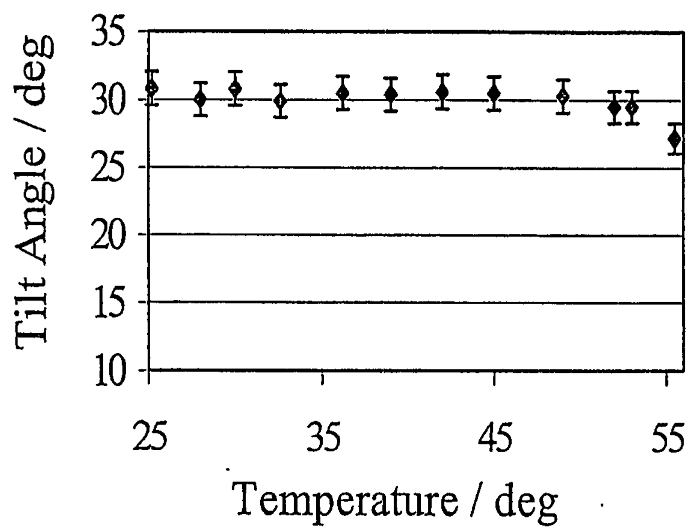


Fig 1 (a)

Saturated switching.

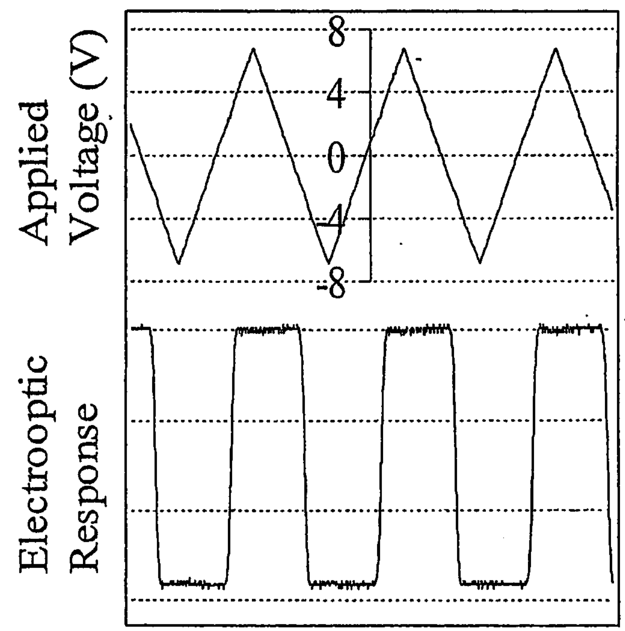


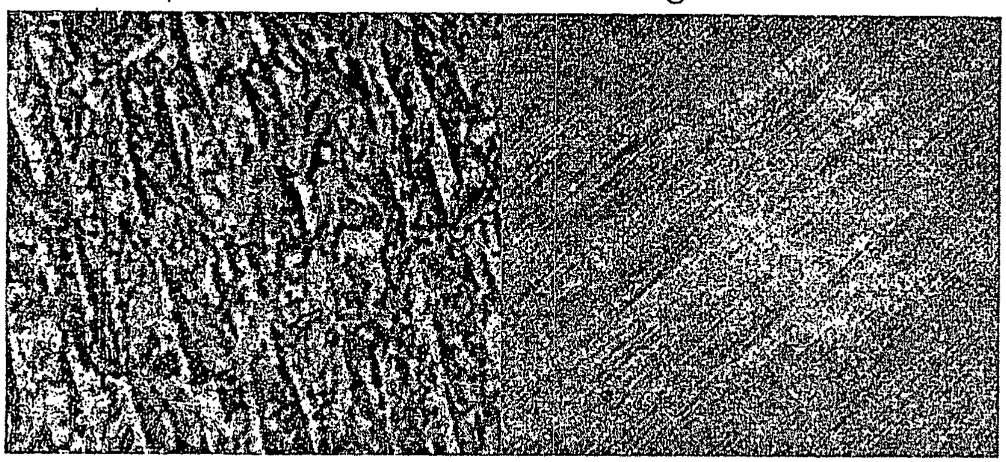
Fig 1 (b)

Bistable Bookshelf Formation with Symmetrical Rectangular Pulses

Siloxane oligomerCDRR8

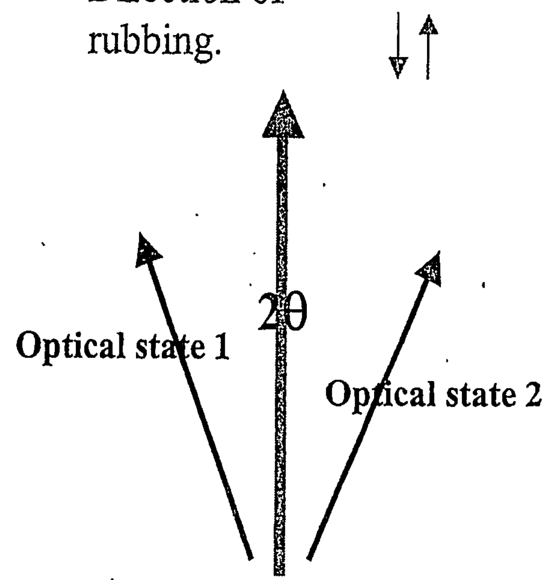
(2.0 μm, nylon 6-6)

Before alignment Fig 2(a) **After alignment**



CS2005 Fig 2(b)
2.81 μm,
nylon 6-6

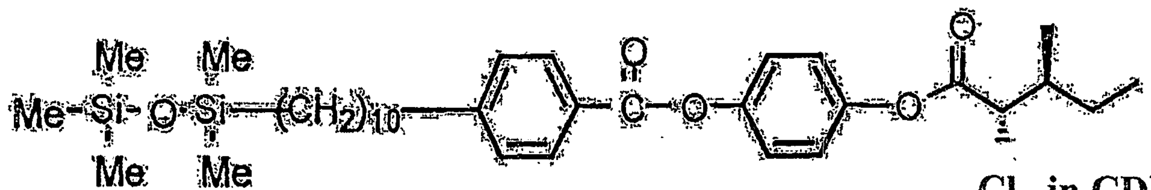
Direction of rubbing.



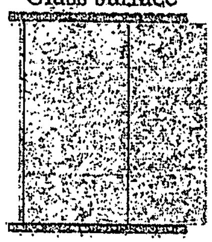
θ is the effective tilt angle

Fields of approximately 10 V/μm and freq. of 1-10 kHz

Bistable Switced States of
Chiral Smectic C Experimental Siloxane Oligomers CDDR3 and CDDR8



Cl in CDDR 3
F in CDDR8



mp = 4.04 °C; Iso 52.68 S_C* -19.71 °C K

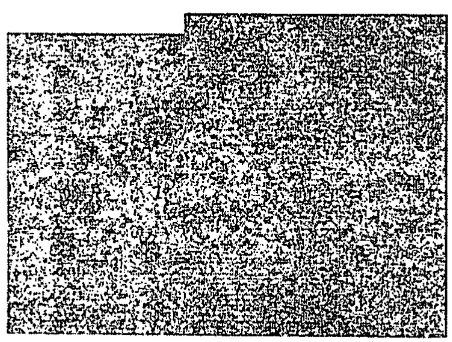
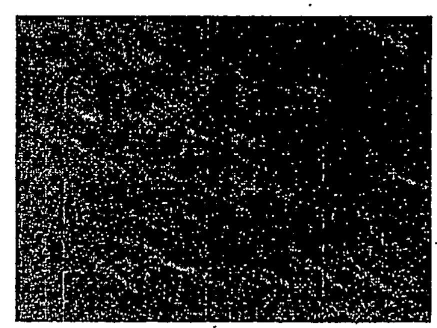


Fig 3 (a)

Fig. 3 (b)



Behaviour suggests decoupling from surface due to long siloxane chains.
This is also suggested by layer rotation.

Asymmetric Waveforms used for Layer Rotation (KHz)

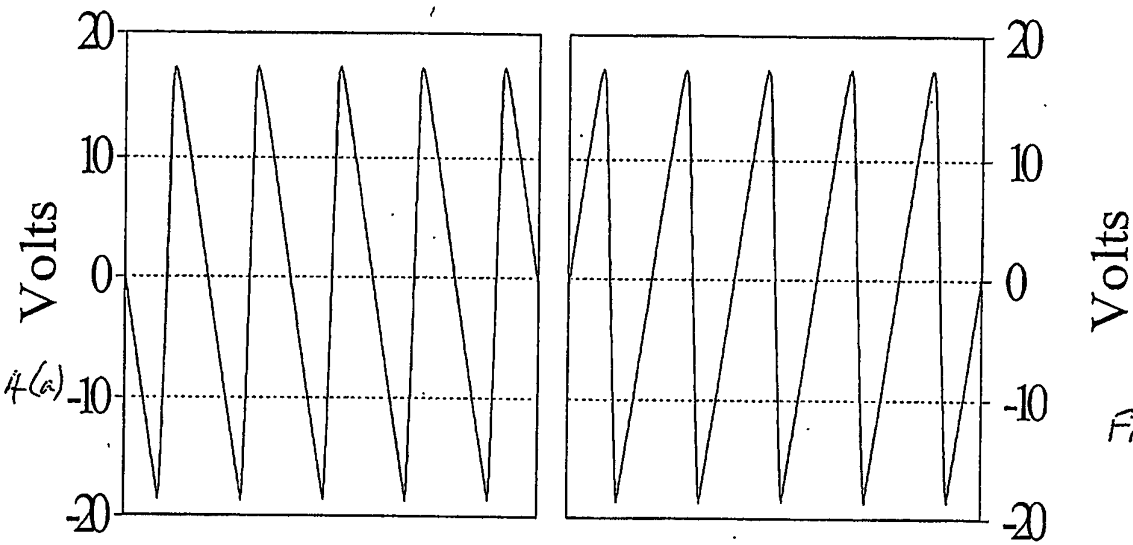


Fig 4(a) Fig 4 (b)

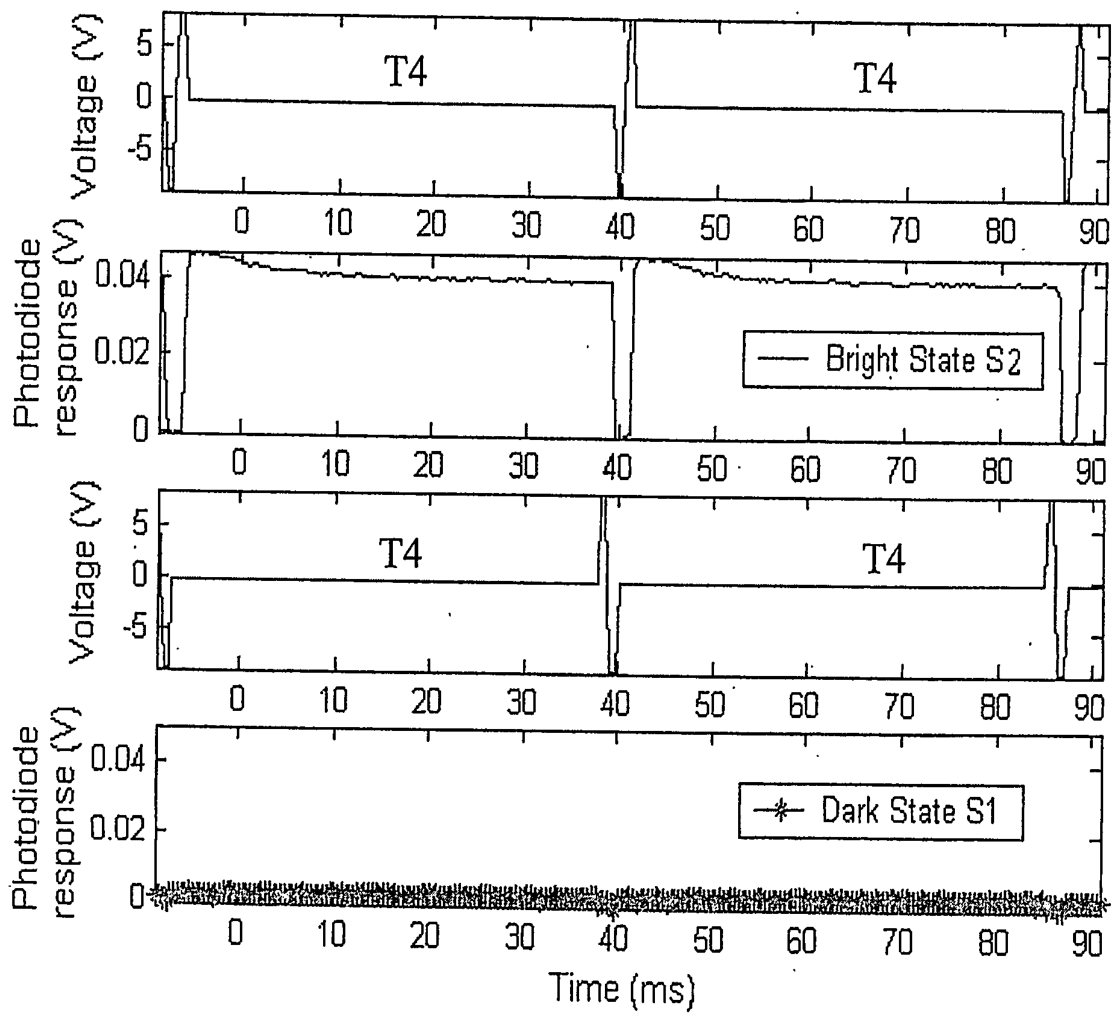
(a) (b)

Right Layer Hand Rotation Left Layer Hand Rotation

IG MANOLIS, MM REDMOND, WA CROSSLAND, AB DAVEY, TD WILKINSON, "Control of the electro-optic bistability of some FLCs useful for binary phase optical modulators", *Mol. Cryst. Liq. Cryst*, 351, 305, 2000

K. Myojin, H. Moritake, M. Ozaki, K. Yoshino, T. Tani, and K. Fujisawa, Jpn. J. Appl. Phys., 33, 5491, (1994).

2.4 Bistability II



Negative-Positive

Fig 5(a)

Fig 5(b)

Value of $I_{0\text{-crossed}}$ at the end of T4

Positive-Negative

Fig 5(c)

Fig 5(d)

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